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 TITLE: 7,2',4'-Trimethoxyflavone
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AB In an attempt to synthesize 2,2'-dihydroxy-4,4'-dimethoxydibenzoylmethane (I) to obtain the spectral characteristics, the intermediate 2,2',4,4'-tetramethoxydibenzoylmethane (II) was cleaved with HI by way of the transitory 2-hydroxy-2',4,4'-trimethoxydibenzoylmethane (III) to the title compound (IV). Polyphosphoric acid (400 g.) and 41.4 g. m-(MeO)2C6H4 stirred 3 hrs. at 50-3° in 21.6 g. AcOH and the mixture diluted with 1200 ml. ice H2O, extracted with Et2O and the dried (Na2SO4) extract evaporated yielded 86.1% 2,4-(MeO)2C6H3COMe (V), b3 134-7°, m. 40-3°. Me2SO4 (1675 g.) and 612 g. NaOH in 1300 ml. H2O used in the treatment of 154 g. β-resorcylic acid according to Robinson and Venkataraman (CA 23, 2181) yielded 80% 2,4-(MeO)2C6H3CO2H, m. 99-104°, esterified (126 g.) in 277 ml. C6H6 with 127 g. alc. in the presence of 3 ml. 100% H2SO4 to yield 89 g. 2,4-(MeO)2C6H3CO2Et (VI), b3.5 143-7°. Freshly prepared NaNH2 (0.5 mole) freed from NH3 and taken up simultaneously in anhydrous Et2O, cooled (solid CO2) and treated in 10 min. with 45 g. V in 42 ml. Et2O, kept 5 min. and treated in 10 min. with 52.5 g. VI in Et2O, the mixture refluxed overnight and quenched in ice H2O containing HCl, the Et2O layer dried and evaporated and the reddish yellow solid recrystd. yielded 37.2% yellow crystalline II, m. 131-4°, λ 6.04, 6.24 μ (CCl4). P2O5 (1.2 g.) and 5.1 g. 85% H3PO4 treated at 20° with 5.0 g. finely powdered KI and 1.7 g. II in succession and the mixture stirred 30 min. at 105-10°, poured into ice H2O and the precipitate recrystd. from dilute alc. yielded 75% IV, m. 143.5-5.5°, λ 6.11 μ (CCl4), λ 236, 334 mμ (ε 2200, 2450, MeOH); oxime, m. 204-7°. Attempts to demethylate II to I by means of AlCl3 or 48% HBr failed, though the transitory III may have formed and given IV by a 1,3-prototropic shift of a methylene H atom, followed by cyclodehydration.

IT 101734-73-0P, Flavone, 2',4',7-trimethoxy-, oxime
 RL: PREP (Preparation)
 (preparation of)

RN 101734-73-0 CAPLUS

CN Flavone, 2',4',7-trimethoxy-, oxime (6CI) (CA INDEX NAME)

